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IS 11720-5 (1993): Methods of test for synthetic rubber, Part 5: Determination of ash [PCD 13: Rubber and Rubber Products]



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“Knowledge is such a treasure which cannot be stolen”

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IS 11720 (Part 5) : 1993

भारतीय मानक

संश्लिष्ट रबड़ की परीक्षण पद्धतियाँ

भाग 5 भस्म ज्ञात करना

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 5 DETERMINATION OF ASH

UDC 578.7 : 543 : 822

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The concerned committee has decided to prepare common methods of test for synthetic rubber under SR (Synthetic Rubber) series, namely, IS 11720 and this will be applicable to all types of synthetic rubbers being produced indigenously. This standard (Part 5) is the fifth in the series. The other standards of this series are as follows:

Part 1 Methods of test for synthetic rubber : Part 1 Determination of antioxidants (SR : 1)

Part 2 Methods of test for synthetic rubber : Part 2 Measurement of vulcanization characteristics with oscillating disc curemeter (SR : 2)

Part 3 Methods of test for synthetic rubber : Part 3 Determination of mooney viscosity and

Part 4 Methods of test for synthetic rubber : Part 4 Determination of volatile matter

In preparation of this standard, considerable assistance has been derived from ISO/DIS 247 'Rubber — Determination of ash' issued by the International Organization for Standardization (ISO).

In reporting the results of a test or analysis made in accordance with this standard, if final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 5 DETERMINATION OF ASH

1 SCOPE

1.1 This standard prescribes two methods for the determination of ash from raw synthetic rubbers, compounded rubbers and vulcanizates.

This standard does not cover the interpretation of the ash results as to the inorganic chemical content of a compound or the vulcanizate. This is the responsibility of the analyst, who has to be aware of the behaviour of rubber additives at elevated temperatures.

1.2 Method A should not be used for compounded or vulcanized rubbers containing chlorine, bromine or iodine.

1.3 Method B should not be used for uncompounded rubbers.

1.4 Lithium and flourine compounds react with silica crucibles to form volatile compounds, giving low ash results. Thus platinum crucibles have to be used for ashing flourine containing and lithium polymerized rubbers.

1.5 The two methods of ashing do not give identical results in all cases, hence it is necessary to state in the test report the method of ashing employed.

2 REFERENCES

The Indian Standards listed below are the necessary adjuncts to this standard:

IS No.	Title
11720 (Part 4) : 1993	Methods of test for synthetic rubber : Part 4 Determination of volatile matter
5599 : 1970	Rubber, raw natural and synthetic — Methods for sampling and sample preparation (<i>first revision</i>)

3 OUTLINE OF THE METHOD

3.1 Method A

Heating of weighed test portion in a crucible over a gas burner. After expulsion of the volatile decomposition products, transfer of the crucible to a muffle furnace where it is heated until all the carbonaceous matter has been burnt off and constant mass is attained.

3.2 Method B

Heating of a weighed test portion in a crucible in the presence of sulphuric acid, first by

means of a gas burner and then in a muffle furnace until all the carbonaceous matter has been burnt off and constant mass is attained.

4 REAGENT

Sulphuric Acid — Analytical grade, 1.84 g/ml (*w/v*).

5 APPARATUS

5.1 Crucible

Porcelain, silica or platinum, of capacity approximately 50 ml. For raw synthetic rubbers, it is permitted to use a crucible of minimum capacity 25 ml per gram of test portion or an aluminium dish or a basin of approximate capacity 50 ml.

5.2 Heat-Resistant, Thermally Insulating Board

100 mm square and of thickness approximately 5 mm, with a central hole to accommodate the crucible. About two-thirds of the crucible shall project below the board.

5.3 Bunsen Burner

Or similar type of gas burner.

5.4 Muffle Furnace

Fitted with a flue and with provision for controlling the air flow through the furnace. (This may be achieved by adjusting the door opening.) A temperature-controlling device is required to maintain a temperature of $550 \pm 25^\circ\text{C}$ or $950 \pm 25^\circ\text{C}$.

6 PREPARATION OF THE TEST PORTION

6.1 Test portions of raw synthetic rubbers shall be cut from the dried rubber obtained after carrying out the determination of volatile matter content in accordance with IS 11720 (Part 4) : 1993.

6.2 Test portions of rubber compounds shall be comminuted by hand.

6.3 Test portions of vulcanizates shall be sheeted or crumbed on a mill or comminuted by hand.

6.4 Care shall be taken to ensure that test portions of rubber compounds and vulcanizates are representative of the sample.

7 PROCEDURE

7.1 Method A

Heat the clean empty crucible of appropriate size for about 30 minutes in the muffle furnace maintained at $550 \pm 25^\circ\text{C}$, allow to cool to ambient temperature in a desiccator and weigh to the nearest 1 mg. Take a test portion of about 5 g of raw rubber or 1 to 5 g of compound or vulcanizate, according to the mass of ash to be expected, and weigh to the nearest 1 mg. Place the weighed test portion in the crucible mounted in the hole in the heat-resistant, thermally insulating board. Heat the crucible gently with the burner, taking care that the rubber does not ignite. If any material is lost due to spurting or frothing, repeat the above procedure with a new test portion.

When the rubber has decomposed to a charred mass, gradually increase the heat until the volatile decomposition products have been substantially expelled and a dry carbonaceous residue remains. Transfer the crucible and its content to the muffle furnace, maintained at $550 \pm 25^\circ\text{C}$, leaving the door of the furnace slightly open to provide sufficient air to oxidize the carbon.

Continue heating until the carbon is completely oxidized and a clean ash is obtained. Remove the crucible and its contents from the furnace, allow to cool to ambient temperature in the desiccator and weigh to the nearest 0.1 mg for uncompounded rubbers. Then heat the crucible and its contents again for about 30 minutes in the muffle furnace, maintained at $550 \pm 25^\circ\text{C}$, allow to cool to ambient temperature in the desiccator and re-weigh to nearest 0.1 mg for uncompounded rubbers, or 1 mg for compounded rubbers. This mass should not differ from the previous mass by more than 0.1 mg in the case of raw rubbers or by more than 1 percent relative to the amount of ash for compounds and vulcanizates. If this requirement is not fulfilled, repeat the heating, cooling and weighing procedure until the difference between two successive weighings meets this requirement.

NOTE — For compounds and vulcanizates, a temperature of $950 \pm 25^\circ\text{C}$ may be used. If this temperature is used, aluminium dishes and basins must not be used and the temperature shall be indicated in the test report along with the reason for its use.

7.2 Method B

Heat the clean empty crucible of appropriate size for about 30 minutes in the muffle furnace, maintained at $950 \pm 25^\circ\text{C}$, allow to cool to ambient temperature in a desiccator and weigh to the nearest 1 mg. Take a test portion of about 1 to 5 g of the compound or vulcanizate

and weigh to the nearest 1 mg. Pour about 3.5 ml of the concentrated sulphuric acid over the test portion to wet the rubber completely. Place the crucible and its contents in the hole on the heat-resistant, thermally insulating board and heat gently with the burner. If, during the initial reaction, the mixture swells excessively, withdraw the flame to avoid possible loss of material.

When the reaction becomes more gentle, increase the heat from the burner until the excess sulphuric acid is volatilized and a dry, carbonaceous residue remains. Transfer the crucible and its contents to the muffle furnace, maintained at $950 \pm 25^\circ\text{C}$, and heat for about 1 h until all the carbon is completely oxidized and a clean ash is obtained. Remove the crucible and its contents from the furnace, allow to cool to ambient temperature in a desiccator and weigh to the nearest 1 mg. Then heat the crucible and its contents again for about 30 minutes in the muffle furnace, maintained at $950 \pm 25^\circ\text{C}$, allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 1 mg.

If this mass differs from the previous mass by more than 1 percent relative to the amount of ash, repeat the heating, cooling and weighing procedure until the difference between two successive weighings is less than 1 percent relative to the amount of ash.

8 EXPRESSION OF RESULTS

Calculate the ash content of the rubber, as a percentage by mass, by the following formula:

$$\text{Ash, percent by mass} = \frac{M_2 - M_1}{M_0} \times 100$$

where

M_0 = mass, in grams, of the test portion;

M_1 = mass, in grams, of the empty crucible;
and

M_2 = mass, in grams, of the crucible and ash.

9 TEST REPORT

The test report shall include the following particulars:

- a) All details required for the full identification of the piece or sample;
- b) Reference to this Indian Standard;
- c) Method employed — A or B;
- d) Temperature used and reason for its choice if 950°C is used for Method A;
- e) Ash of the product tested, as a percentage by mass; and
- f) Date of the test.

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